RADIATION DEFECTS IN NaCl MATRIX WITH REDUCED LATTICE SYMMETRY CAUSED BY LIGHT CATION DOPING AND ELASTIC UNIAXIAL DEFORMATION

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The processes of radiation defect creation and radiative relaxation of electronic excitations under applied local or/and uniaxial elastic deformation have been studied in NaCl crystals by means of optical absorption, luminescence and thermoactivation spectroscopy methods. In NaCl:Li at 80 K, X-ray-induced absorption bands peaked around 3.35 and 4.6 eV have been detected and ascribed to interstitial halide atoms located nearby Li impurity cations, H_A(Li) centres. Subsequent thermal annealing of H_A(Li) centres leads to the formation of polyhalide centres responsible for the absorption band at 5.35 eV. In an X-irradiated and stressed NaCl:Li crystal (degree of uniaxial elastic deformation of $\varepsilon = 0.9\%$), the peak of thermally stimulated luminescence at ~115 K is composed of the ~2.7-eV emission appearing, in our opinion, due to the recombination of the electron, thermally released from an F' centre, with a hole-type H_A(Li) centre. The applied uniaxial elastic stress facilitates the self-trapping of anion excitons in regular regions of a NaCl lattice and impedes the energy transfer by mobile excitons to impurities/defects and, in turn, attenuates the Br-related luminescence peaked at 3.95 eV with respect to the π-emission of self-trapped excitons (~3.35 eV). The 3.95 eV emission has been detected in a natural NaCl crystal containing homologous Br impurity ions.

Keywords: alkali halide crystal, self-trapped exciton, electronic excitation, uniaxial elastic deformation

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1. Introduction

For a long time, alkali halide crystals (AHCs) have served and are still serving as model systems with a rather simple electron/crystal structure in studies of various physical processes in wide-gap solids. The concept of electronic excitations (EEs) which are conduction electrons and valence holes, as well as mobile and current-free excitons, was successfully applied in these studies.

Particular emphasis was paid to the evolution of anion excitons formed directly by photons with the corresponding energy or via the recombination of electron–hole pairs, in particular, to their migration though a crystal lattice, self-trapping (complete loss of mobility) in regular lattice regions or localization near the existing imperfec-tions (see [1–13] and references therein). It is generally accepted that, besides ordinary heat release, two other energy dissipation channels of anion self-trapped/trapping excitons (STEs) in AHCs are (i) radiative annihilation with the emission of a quantum of intrinsic luminescence and (ii) non-radiative decay with the formation of vacancy-interstitial Frenkel defects − the so-called $F–H$ and $\alpha–I$ pairs of anion defects [2, 3, 11, 14–27]. The relative efficiency of these channels should be taken into account developing various functional optical materials (scintillators, dosimeters, materials for lighting, tolerant to harsh irradiation, etc.). Note that the majority of anion Frenkel pairs have a very short lifetime $10^{-11}–10^1$ s (the so-called short-lived defects with a small distance between complementary defects in the pairs), while only about

5% of interstitial-vacancy Frenkel defect pairs are stable for days and months [2, 14–27, 20–24, 27]. Just these stable structural defects, all of which are totally immobile only at helium temperatures and gradually become mobile at crystal heating (see, e.g. Refs. [14, 19, 20]), are the subject of the present study.

At present, the features of different stages of EE (anion exciton) relaxation/localization in AHCs that are doped with various anion/cation impurity ions and contain vacancy-type defects, both as-grown ones and induced via previously applied plastic deformation, have been thoroughly studied (see, e.g. Refs. [28–33]). Meanwhile, the processes of EE relaxation under continuous external impact (e.g. uniaxial elastic stress or hydrostatic pressure) and reduced crystal lattice symmetry are studied less. Nevertheless, it was established, in particular, that the lowering in the symmetry of the AHC lattice led to the decrease in a free path of an anion exciton, increased the probability of exciton self-trapping at regular lattice sites and, in turn, caused the enhancement of intrinsic STE luminescence [23, 34–38].

The present study is devoted to the mechanisms of radiation creation, recombination, and thermal destruction of point structural defects under conditions of reduced crystal lattice symmetry resulting from material doping with light/small-radius impurity cations or applied low-temperature elastic uniaxial deformation. This study was performed using NaCl and NaCl:Li crystals as an example and by means of absorption, luminescence and thermoactivation spectroscopy methods.

2. Experiment

The X-ray luminescence (XRL) spectra of crystals were recorded using a RUP-120 X-ray unit operating in the 3 mA and 100 kV mode. Note that the used hard X-ray radiation penetrates the sample throughout its entire thickness and does not create radiation defects, which may cause reabsorption in the corresponding regions of the emission spectrum. The latter circumstance is important, because the luminescence band of exciton-like formations with a maximum at 2.7 eV in NaCl matrix practically coincides with the absorption band of the *F* centre (an electron trapped in the field of

anion vacancy). Special experiments have shown that the X-ray radiation required to record the XRL spectra at 90 K did not lead to the accumulation of a detectable *F*-absorption at 2.7 eV, and, in turn, the measured spectra were not distorted by reabsorption.

The optical absorption spectra of the crystals irradiated with X-rays at 80 K were recorded by an Evolution-300 spectrophotometer, while a background from an irradiated crystal was automatically subtracted. Therefore, the spectra presented in this work characterize the optical absorption induced by X-ray irradiation (more specifically, the optical density OD).

The experimental setup allows scanning in a range of 200–850 nm the spectra of XRL and thermally stimulated luminescence (TSL), as well as measuring of the integral TSL curves of the crystals subjected to the elastic uniaxial deformation at 90 K. The measurements were carried out using an MSD-2 high-aperture monochromator with an H 8259 photomultiplier (*Hamamatsu*) operating in the photon counting mode and controlled by special programs SpectraScan and ThermoScan. Low-temperature deformation and recording of the luminescence characteristics of the crystals were carried out using a special cryostat equipped with appropriate windows [39].

When measuring the TSL curve with a constant heating rate (0.15 K/s), the scanning time of each emission spectrum at 2.0–6.0 eV corresponds to the sample temperature changes only by 1.5–2.0 K. Therefore, using the maximum scanning speed (50 nm/s), many emission spectra were recorded within each TSL peak, and it was possible to verify the constancy (or changes) of the spectral composition of thermoluminescence.

The study was carried out on NaCl crystals of high purity (manyfold recrystallization from the melt) and NaCl:Li (0.2 mol%), synthesized at the Institute of Physics, University of Tartu, as well as natural NaCl crystals (halite) grown in natural conditions at Sol-Iletsk deposit, Russia. Unlike crystals synthesized at high temperatures (above 800°C), natural halites were grown at low temperatures (5–10°C) what provides a high level of purity from divalent impurities. At the same time, according to the literature data [26, 40], natural NaCl crystals still contain anion homologues, bromine ions.

3. Absorption spectra of radiation defects in NaCl:Li

Thermally stable radiation defects are formed as a result of the interaction of two mobile interstitial halogen atoms (*H* centres) with the formation of a trihalide X_3^- centre, which is a complementary defect to the *F* centre that remains stable at high temperatures (see, e.g. Refs. [21, 26, 29]). Thus, the creation efficiency of radiation defects in NaCl crystals at relatively high temperatures is determined by the efficiency of X_3^- centre formation.

It is assumed that due to the local deformation of the NaCl lattice by light impurity cations (Li), it becomes possible to monitor the formation efficiency of Cl_3^- centres, both during the crystal irradiation with X-rays at 8 K and at further thermal destruction of H_A(Li) centres (an *H* centre localized near an impurity cation). By analogy with the previously studied doped AHCs (see, for example, Refs. [12, 28, 41]), mobile interstitial halogen atoms, formed during X-irradiation at 80 K, can both interact with each other and become localized in the field of impurity lithium ions (i.e. to form the H_A centres). On

the other hand, only the possibility of pair interaction of mobile halogen atoms resulting in the formation of thrihalide Cl_3^- centres still remains after the total thermal destruction of H_A(Li) centres.

In the optical absorption spectrum of the NaCl:Li crystal irradiated with X-rays at 80 K, two bands with maxima at 3.35 and 4.6 eV, which are absent in a highly pure NaCl crystal, have been detected (see Fig. 1, curve 1). In addition, the concentration of both the *F* and *F'* centres (two electrons in the field of an anion vacancy) is higher in a doped sample. By analogy with the KCl:Li crystal [12], the radiation-induced absorption bands at 3.35 and 4.6 eV in NaCl:Li (inset *a* in Fig. 1), which undergo simultaneous thermal annealing at 130 K (see inset *b* in Fig. 1), are apparently associated with the H_A(Li) centres (see insets in Fig. 1).

Upon the thermal destruction of H_A(Li) centre (~130 K), the crystal lattice becomes rearranged via the formation of trihalide centres with a maximum at 5.35 eV, as a result of pair interaction between becoming mobile interstitial halogen atoms, *H* centres (see Fig. 1, curve 2). Further heating of the irradiated NaCl:Li crystal to room temperature causes
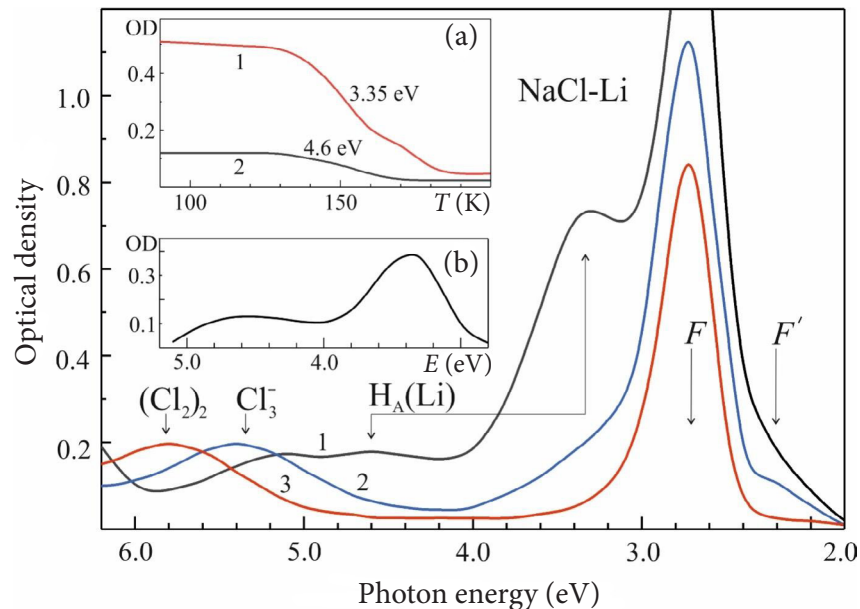

Fig. 1. Absorption spectra measured at 80 K for a NaCl-Li crystal irradiated with X-rays at 80 K: curve 1, immediately after irradiation for 6 h; curves 2 and 3, after additional preheating to 150 and 300 K, respectively. Inset *a* shows thermal annealing of the absorption bands peaked at 3.35 and 4.6 eV. (b) Inset *b* is the absorption spectrum of the H_A(Li) centres obtained as a difference spectrum representing the decay of OD between subsequent preheating to 100 and 160 K.

a subsequent transformation of trihalide quasi-molecules into more complex and thermally stable up to 400 K polyhalide centres (for example, $(X_2)_2$ centres studied in [42]) with a maximum at 5.8 eV (Fig. 1, curve 3). At the same time, the decrease of electron-type F and F' centre concentration is observed due to both the recombination of electrons from these centres with delocalized hole centres and thermal ionization of the F' centres themselves (Fig. 1, curves 2 and 3). In addition, the annealing of one more absorption band with a maximum at high photon energy (above the experimental limit at 6.4 eV) is observed (compare curves 1 and 2 in Fig. 1). Similar to KCl:Li and KCl:Na crystals [12], this band could be tentatively ascribed to the I_A(Li) centres (interstitial halogen ion localized nearby an impurity cation).

Thus, according to our experimental data, the doping of a NaCl crystal with light lithium cations, the ionic radius of which is smaller than that of the matrix cation, leads to localization of mobile interstitial halogen atoms in the form of the H_A(Li) centres (see also Ref. [40]). It is worth noting that the doping of NaCl by lithium ions practically does not affect the efficiency of Cl_3^- centre creation by X-rays at 80 K.

4. Thermally stimulated luminescence related to radiation defects in NaCl:Li

Figure 2(a) demonstrates the TSL curve measured for a spectrally integrated signal with a constant heating rate $\beta = 0.15$ K/s for a NaCl:Li crystal
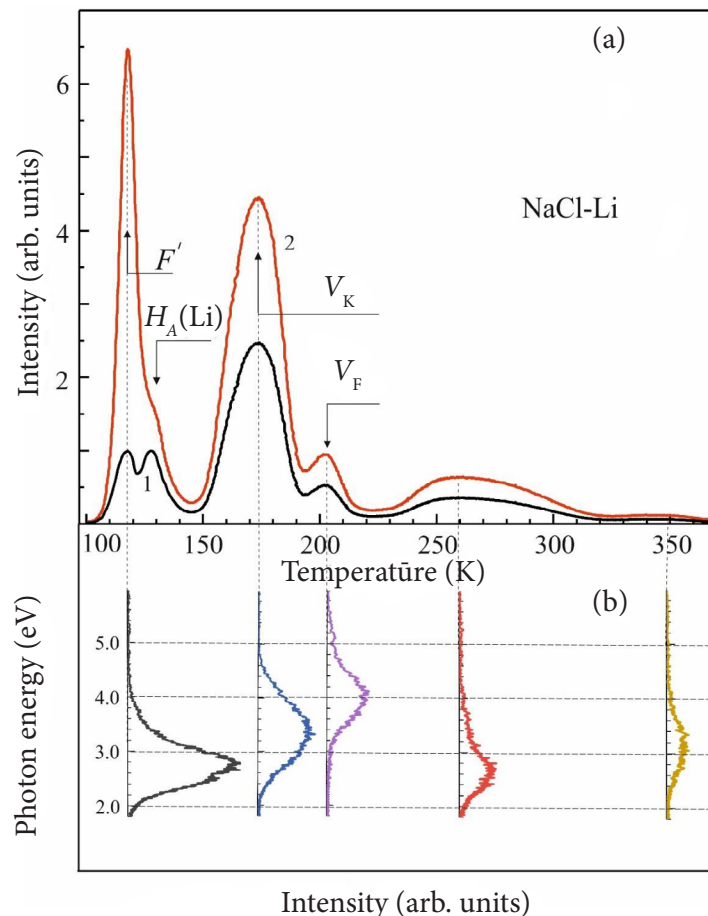

Fig. 2. (a) Curves of spectrally-integrated TSL measured with a constant heating rate of 0.15 K/s for a NaCl:Li crystal previously exposed to X-rays at 90 K (1 h). Curve 2, with additionally applied elastic uniaxial stress ($\varepsilon = 0.9\%$) at 90 K; curve 1, for a non-stressed sample. (b) Emission spectra measured within different TSL peaks of a NaCl:Li crystal X-irradiated and additionally stressed ($\varepsilon = 0.9\%$) at 90 K.

exposed to X-rays at 90 K. TSL consists of several peaks at ~115 K, 130, 170, 205 and 250–260 K connected with a thermal release of carriers from different defects/impurities (curve 1, see also Ref. [40]). The intensity of these peaks is significantly higher (curve 2) if the same crystal (preheated to 500 K, i.e. de-excited) was repeatedly irradiated by the same dose of X-rays but an elastic uniaxial stress was additionally applied at 90 K. Also note that the TSL of the stressed NaCl:Li is more intense than that in a high-purity NaCl crystal.

According to Fig. 2, a low-temperature elastic deformation sharply enhances the TSL peaks at ~115 and ~170 K associated with the F' and V_K centres, respectively [40]. The V_K centre is a self-trapped hole in the form of a quasi-molecule X_2^- occupying two nearest neighbour anion sites of the lattice [43], while an interstitial halogen atom or the H centre could be represented as the same X_2^- located at only one anion site [44]. On the other hand, the intensity of the TSL peak at ~130 K associated with the thermal destruction of the H_A(Li) (confirmed also by EPR data [40]) centres practically remains the same in stressed or unstressed samples. Such behaviour with respect to the subjection to elastic uniaxial stress is typical of impurity-related TSL peaks.

Figure 2(b) presents the emission spectra measured within different TSL peaks of an elastically deformed NaCl:Li crystal. The luminescence with a maximum at 2.7 eV dominates in the temperature region related to thermal ionization of the F' centres (~115 K). Such emission arises due to the radiative recombination of a released conduction electron with a still localized hole-type H_A(Li) centre via the formation of an exciton-like excitation in the field of a light ion lithium (the so-called near-impurity/defect-localized or bound exciton, see, e.g. Refs. [11, 19, 45]). On the other hand, an electron released from the F' centre can alternatively interact with the V_K centre nearby the Li ion. However, such assembly of a two-halide exciton localized near Li undergoes radiative decay with the luminescence at ~3.35 eV [20, 40]. The latter spectrally coincides with the π-component of intrinsic STE emission in NaCl crystals, while its thermal quenching occurs at rather higher temperature (150 and 90 K, respectively) [2, 3, 7, 40].

It is worth noting that the absence of the 2.7 eV emission in the TSL spectra measured above

the delocalization temperature of the H_A(Li) centre (~130 K) in the elastically deformed NaCl:Li crystal clearly indicates that this luminescence is caused by the recombination of conduction electrons appearing due to the thermal ionization of F' centres (~115 K) with hole-type H_A(Li) centres. Although the radiative state of this exciton-like excitation (a bound exciton) resulting in the 2.7 eV emission is not clear yet, in our opinion, it is definitely not a classical two-halide STE located in the field of an impurity ion. There is no information in the literature on a luminescence band at 2.7 eV in NaCl crystals not subjected to external action.

The V_K centre becomes mobile at ~170 K and can recombine with an electron from the still immobile F centre. As a result of such radiative recombination, both the 3.4-eV STE luminescence at regular lattice sites and the emission with unresolved maxima of about 2.9 and 3.95 eV connected with exciton self-trapping in the field of vacancy-type defects can be detected (see Fig. 2(b)). The recombination of a V_F centre (V_K centre localized nearby a cation vacancy) that becomes mobile at ~205 K with an immobile electron-type F centre is accompanied by the emission peaked at 3.95 eV, similar to the luminescence of STE located in a divacancy field (see, e.g. Refs. [29–32]).

5. X-ray luminescence of NaCl crystals exposed to elastic uniaxial deformation

Additional information on the nature of the emissions dominating in different TSL peaks could be obtained from XRL spectra measured for NaCl crystals with a different level of purity and additionally subjected to elastic deformation at 90 K.

One of the main types of intrinsic luminescence in NaCl crystals is the STE luminescence, which at 4.2 K consists of two bands with maxima at ~3.35 eV (π-component) and 5.4 eV (σ) [2–4, 7, 8, 18]. These emissions are effectively excited by photons with energies ~8 eV (exciton absorption peak) and $h\nu > 8.8$ eV (a start of interband transitions), corresponding to the creation of excitons and electron–hole pairs (see, e.g. Refs. [6, 7, 11, 14, 25]). The polarization of the STE luminescence components is indicated in parentheses. With an increase in temperature, both luminescence bands are sharply quenched; and their intensity at 90 K

decreases by almost two orders of magnitude with respect to the initial values. Nevertheless, both exciton emission components could be clearly recorded at this temperature in the XRL spectra.

Figure 3(a) (curve 1) shows the XRL spectrum of a high-purity NaCl crystal at 90 K, where noticeably quenched STE emission bands at ~5.3 and 3.4 eV are clearly visible. The elastic uniaxial deformation applied to the crystal (up to $\varepsilon = 0.9\%$) increases the intensity of both emission bands by more than 8 times (compare curves 1 and 2 in Fig. 3(a)). A similar increase (also by a factor of
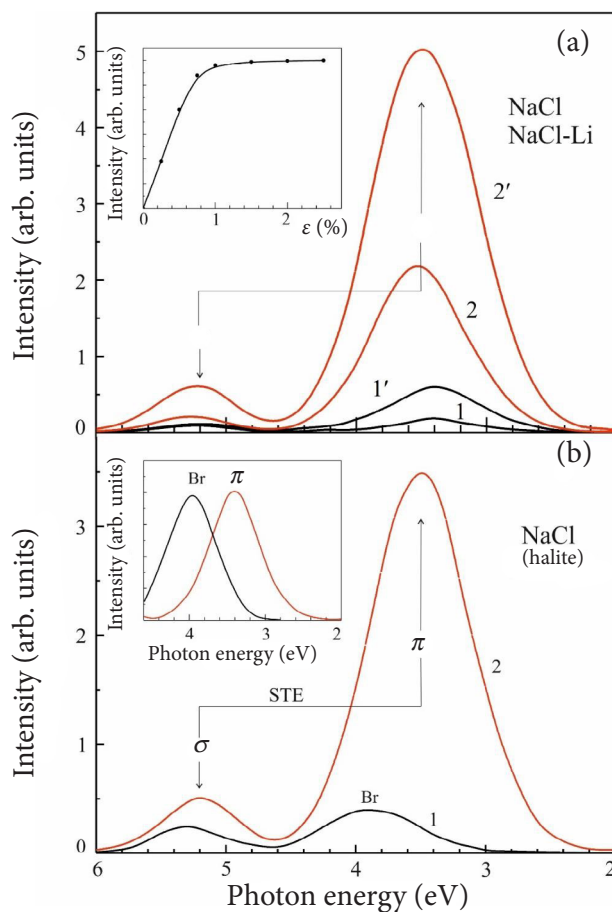

Fig. 3. X-ray luminescence spectra of NaCl crystals at 90 K. (a) XRL spectra for a high-purity NaCl and a NaCl:Li crystal before (curves 1 and 1') and under applied elastic uniaxial stress with $\varepsilon = 0.9\%$ at 90 K (2, 2'). The inset shows the dependence of the π-luminescence intensity (3.4 eV) on the degree of deformation ε. (b) XRL spectra for a non-stressed (curve 1) and stressed ($\varepsilon = 0.9\%$ at 90 K) natural NaCl (halite) crystal. The inset shows normalized low-energy XRL bands for a non-stressed (Br impurity-related emission) and a stressed sample (σ-STE luminescence).

more than 8) in the STE luminescence intensity was detected in XRL of NaCl: Li (0.2 mol%) crystals (compare curves 1' and 2').

Neither additional bands have been detected in XRL spectra of elastically deformed NaCl and NaCl:Li (0.2 mol%) crystals. At the same time, the intensity of the π-STE emission in the stressed NaCl:Li is twice as high as in the stressed high-purity crystal (compare curves 2 and 2' in Fig. 3(a)). Note that the intensity ratio for XRL in the spectral region typical of the π-emission in these non-stressed crystals is practically the same (curves 1 and 1' in Fig. 3(a)). This is apparently due to the fact that the maxima of the STE (3.35 eV) and near-lithium exciton (~3.35 eV [20, 40]) emissions coincide.

In high-purity NaCl and NaCl:Li crystals, the dependence of XRL at ~5.3 and 3.4 eV on the relative deformation degree ε has two stages: the first, linear stage of $I = f(\varepsilon)$ characterizes elastic uniaxial deformation, while the second stage demonstrates saturation of luminescence intensity with ε and characterizes the plastic part of the deformation (see the inset in Fig. 3(a)). A similar dependence $I = f(\varepsilon)$ has been obtained for XRL in a natural NaCl (halite) crystal as well.

In a natural NaCl crystal, two XRL bands with maxima at 3.95 and 5.25 eV have been detected at 90 K (see Fig. 3(b), curve 1). There is a broad emission band peaked at 3.95 eV with an apparently complex structure due to the superposition of several bands, instead of the ~3.35 eV luminescence band in a synthesized, high-purity NaCl crystal. In NaCl (halite), a sharp increase of the π-luminescence intensity with ε takes place causing, in turn, a shift of the complex band maximum from 3.95 eV to lower energies; at $\varepsilon = 1\%$ the maximum becomes located at ~3.4 eV.

The inset in Fig. 3(b) demonstrates the normalized low-energy XRL bands of a natural NaCl crystal before and during elastic deformation. In a non-stressed crystal, the Br-related luminescence is dominant, while such impurity-related emission is absent in a high-purity crystal. The ~3.95 and 5.25 eV XRL emissions of the non-stressed natural NaCl (Fig. 3(b), curve 1) belong to the STEs (π and σ components). The applied elastic deformation enhances the intensity of these XRL bands by more than 8 times. In addition, the XRL of the stressed natural crystal (Fig. 3(b), curve 2)

practically coincides with that in the synthesized, high-purity NaCl and contains the π (~3.4 eV) and σ (5.35 eV) emission components of STEs in a regular lattice.

6. Concluding remarks

On the basis of numerous experimental data [33–36], the conception of the influence of the applied elastic deformation on the luminescence characteristics of AHCs was formulated as follows. The applied elastic uniaxial stress increases the probability of the self-trapping of mobile anion excitons at regular lattice sites, and subsequent radiative relaxation of STEs results in the typical intrinsic luminescence of AHCs [37–38].

This conclusion was firstly based on the experimentally detected redistribution of the intensity of impurity luminescence (thallium-related, peaked at 2.85 eV) in favour of the π-STE emission (3.3 eV) in a KI:Tl crystal exposed to elastic deformation [46]. This enhancement of the intrinsic luminescence of KI:Tl with an elastic deformation degree at 90 K was interpreted as a consequence of the reduction in the free path of the mobile anion exciton before their self-trapping. Thus, the lowering of AHC lattice symmetry by the applied uniaxial elastic deformation causes a sharp decrease (by more than 70 times) of the mean free path of EEs (anion excitons), and in turn facilitates the self-trapping of anion excitons in regular lattice regions and impedes the energy transfer by mobile excitons to impurities and structural defects. As a result, the deformed crystal exhibits significant weakening of the luminescence associated with the presence of impurity ions and structural (i.e. vacancy-type) defects.

In the present study, the influence of both the applied elastic uniaxial deformation and the local deformation of the NaCl lattice by light cation doping has been analysed. In X-irradiated NaCl:Li, two radiation-induced absorption bands at 3.35 and 4.6 eV at 80 K were attributed to near-impurity-localized interstitial halogen atoms, the H_A(Li) centres. Thermal destruction of these H_A(Li) centres starts at about 130 K and causes the crystal lattice rearrangement via the formation of Cl_3^- centres with a typical absorption band at 5.35 eV. At room temperature, these trihalide quasi-molecules undergo transformation into more complex, stable up to 400 K polyhalide centres responsible for optical absorption with a maximum at 5.8 eV.

In X-irradiated NaCl:Li with a simultaneous action of the elastic uniaxial stress ($\varepsilon = 0.9\%$) and Li-related local deformation, the emission spectra within TSL peaks below 130 K contain a band with a maximum at 2.7 eV, which, in our opinion, could be interpreted as a radiative recombination of the conduction electrons released via thermal ionization of the F' centres with the hole-type and still immobile H_A(Li) centres. In a natural NaCl (halite) crystal, the applied elastic deformation suppresses the luminescence band 3.95 eV connected with impurity bromine ions.

It was demonstrated that along with the weakening of impurity emissions, the applied elastic deformation enhances the intrinsic luminescence of the crystal. This circumstance makes it possible to use elastic deformation as the experimental method for separating the intrinsic and impurity luminescence of crystals. Recently, we have already applied this method to confirm the intrinsic nature (exciton-like luminescence) of the so-called E_x emission (3.1 eV) in RbI crystals (see [47]).

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RADIACINIAI DEFEKTAI NaCl MATRICOJE SU DĖL NEDIDELIŲ KATIJONINIŲ PRIEMAIŠŲ IR ELASTINĖS VIENAAŠĖS DEFORMACIJOS SUMAŽINTA GARDELĖS SIMETRIJA

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